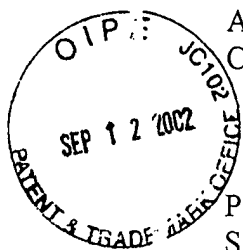


Attorney Docket No. 1700.109
Confirmation No. 2417

PATENT



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Pillai et al.
Serial No. 09/977,127
Filed: October 12, 2001
For: HETEROGENEOUS Pd CATALYSTS AND
MICROWAVE IRRADIATION IN HECK ARYLATION

Group Art Unit: 1711

Box Missing Parts
Assistant Commissioner for Patents
Washington, D.C. 20231

May 13, 2002

PETITION TO FILE ON BEHALF OF UNREACHED JOINT INVENTORS

Sir:

This is a petition under 37 CFR 1.47 to file in the name of joint inventors whom to date have not been reached after diligent effort. The Notice of Missing Parts was mailed on November 13, 2001. Thus, Applicants are required to respond by the date of this petition (May 13, 2002). A petition for an extension of time of four (4) months to file the missing parts is enclosed herewith.

The fee (\$130.00) for this petition under 37 CFR 1.17(h) is also included.

The named inventors are A. Wali, S.M. Pillai, and S. Satish. The executed Declaration of joint inventor S. Satish is enclosed. The unreachable (to date) inventors are S.M. Pillai and A. Wali.

The last known addresses of the unreachable joint inventors are as follows:

S.M. Pillai
IPCL
PO: Petrochemicals 391346
Vadodara
India

RECEIVED

OCT 07 2002

OFFICE OF PETITIONS

Pillai et al.
Serial No. 09/977,127
Filed: October 12, 2001
Page 2

A. Wali
IPCL
PO: Petrochemicals 391346
Vadodara
India

Applicant Satish expects that inventors Pillai and Wali will eventually be reached. To date, however, they have not responded to diligent efforts to contact them and have them execute their declarations.

Inventors Pillai and Wali are residents of India. The diligent efforts to date include:

- attempts to gain assistance in India using legal counsel in the United Kingdom;
- attempts to contact the inventors using legal counsel in India;
- attempts by a U.S. visitor to meet the inventors in person;
- attempts to reach the inventors via e-mail; and
- attempts to reach the inventors using the Global Express Mail service of the United

States Postal Service (USPS);

Attempts at contacting the inventors began as early as April 2001 (Exhibits 1 and 2). This included contact directly from undersigned counsel and from an interested corporate party in a related area of technology (Exhibit 3). This contact extended in an initial phase through May 2001 (Exhibits 4 and 5).

In August 2001, undersigned counsel requested assistance from counsel in the United Kingdom for the purpose of identifying and contacting the inventors (Exhibits 6 and 7). Based on the information received from counsel in the United Kingdom, undersigned counsel conducted further inquiries through a law firm in India (Exhibit 8). This contact consisted of several items of correspondence during September of 2001 (Exhibits 9, 10, 11 and 12). As a method of confirming receipt, Exhibit 9 was sent by secure electronic courier and Exhibit 10 (which is an identical letter) by facsimile.

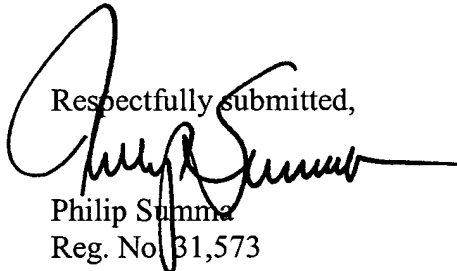
Undersigned counsel continued to follow up with counsel in India during December 2001 (Exhibit 13) and into January of 2002 (Exhibits 14 and 15).

Pillai et al.
Serial No. 09/977,127
Filed: October 12, 2001
Page 3

Some information became available in February of 2002 (Exhibit 16) and in April of 2002 (Exhibits 17 and 18).

On April 24, 2002, undersigned counsel sent declarations to named inventors Wali and Pillai in individual packages using the Global Express Mail service of the USPS (Exhibits 19 and 20). Applicants have continued to check on the status of delivery of these materials from April 24, to date (Exhibits 21 and 22) but without success. The notes made by the assistant to undersigned counsel are included as Exhibit 23 and demonstrate repeated attempts to track these packages from April 24, 2002 through May 13, 2002.

Accordingly, the deadline for filing the declarations with appropriate extensions now having arrived, the declaration of inventor Satish is enclosed along with this petition that the examination of the application proceed until such time as the unreached inventors submit their declarations.

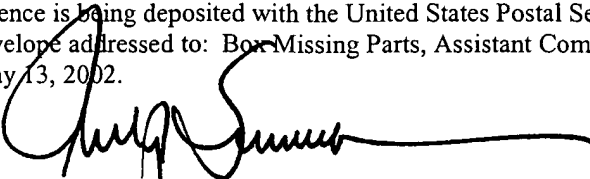
Respectfully submitted,

Philip Summa
Reg. No. 31,573

021176
Summa & Allan, P.A.
11610 North Community House Road
Suite 200, Ballantyne Corporate Park
Charlotte, North Carolina 28277-2162
Telephone: 704/945-6701
Facsimile: 704/945-6735
S:\FIRM DOCS\1700\109\Petition051302.doc

Pillai et al.
Serial No. 09/977,127
Filed: October 12, 2001
Page 4

CERTIFICATE OF MAILING

I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to: Box Missing Parts, Assistant Commissioner for Patents, Washington, D.C. 20231, on May 13, 2002.


Philip Summa, Reg. No. 31,573



List of Exhibits

Exhibit No.	Item	Date
1	Facsimile letter from Philip Summa to Drs. Wali, Pillai, Satish	April 4, 2001
2	Email message from Philip Summa to IPCL	April 4, 2001
3	Letter to Dr. Ravindranath at IPCL	April 9, 2001
4	Email message from Applicant to Dr. Pillai	May 3, 2001
5	Facsimile letter from Dr. Pillai	May 24, 2001
6	Facsimile letter from Philip Summa to counsel in United Kingdom	August 23, 2001
7	Facsimile letter from counsel in United Kingdom to Philip Summa	August 29, 2001
8	Electronic courier letter from Philip Summa to counsel in India	August 30, 2001
9	Electronic courier letter from Philip Summa to counsel in India	September 6, 2001
10	Facsimile letter from Philip Summa to counsel in India	September 11, 2001
11	Facsimile letter from counsel in India to Philip Summa	September 12, 2001
12	Facsimile letter from Philip Summa to counsel in India	September 12, 2001
13	Electronic courier letter from Philip Summa to counsel in India	December 10, 2001
14	Letter via international courier service from Philip Summa to counsel in India	January 11, 2002
15	Facsimile letter from counsel in India to Philip Summa	January 16, 2002
16	Facsimile letter from counsel in India to Philip Summa	February 1, 2002
17	Email message from Dr. Satish	February 6, 2002
18	Email message from David Fish to Cindy Moser	April 9, 2002
19	Copy of Global Express Mail receipt No. EU423804792US	4/24/02 at 10:10 a.m.
20	Copy of Global Express Mail receipt No. EU423804789US	4/24/02 at 10:10 a.m.
21	Copy of Internet delivery status for Global Express Mail No. EU423804792US	May 13, 2002
22	Copy of Internet delivery status for Global Express Mail No. EU423804789US	May 13, 2002
23	Diary of Global Express Mail delivery status for EU423804792US and EU423804789US	April 24, 2002 through May 13, 2002



Philip Summa, P.A.
13777 Ballantyne Corporate Place
Suite 315
Charlotte, NC 28277
704-945-6700
Facsimile: 704-945-6735

VIA FACSIMILE: 011-91-265-375-165

Dr. A. Wali
Dr. S. Muthukumar Pillai
Dr. S. Satish
Research Center
Indian Petrochemicals Corporation Ltd.
Vadodara-391 346
INDIA

Dear Sirs:

I am a patent attorney in the United States. I am investigating the use of microwave irradiation in the Heck reaction. My research has led to your article published in React. Catal. Lett., Volume 60, Number 1, Pages 189-194 (1997) on this subject.

In your article you state that yours was the first use of microwave irradiation in Heck reactions. I would appreciate knowing if your work was published (or presented orally, etc.) any earlier than 1997, including in any country or any language.

By way of background, a United States patent was recently issued covering the use of microwave irradiation in the Heck reaction. Based upon my research, the work of the alleged inventors appears to be at best concurrent with, and possibly later than, your work. We are investigating the validity of that patent, and any evidence of work done concurrently or earlier by others would be an important part of our evaluation.

Therefore, we would accordingly appreciate any assistance you could provide in directing us to publications (or other presentations) for which you or others are the authors that deal with microwave irradiation of the Heck reaction, particularly in 1995 or earlier years.

Thank you for your assistance.

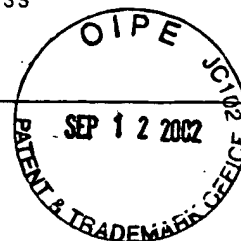
Very truly yours,

Philip Summa

RECEIVED

OCT 07 2002

OFFICE OF PETITIONS



TRANSACTION REPORT

Transmission

ERROR: Re-send error page(s).

NO.	TX DATE/TIME	DESTINATION	DURATION	PGS.	RESULT	MODE
211	APR. 4 13:01	01191265270040		000	BUSY	



Philip Summa, P.A.
13777 Ballantyne Corporate Place
Suite 315
Charlotte, NC 28277
704-945-6700
Facsimile: 704-945-6735

VIA FACSIMILE: 011-91-265-⁷¹⁰~~370~~-040

Dr. A. Wali
Dr. S. Muthukumar Pillai
Dr. S. Satish
Research Center
Indian Petrochemicals Corporation Ltd.
Vadodara-391 346
INDIA

Dear Sirs:

I am a patent attorney in the United States. I am investigating the use of microwave irradiation in the Heck reaction. My research has led to your article published in React. Catal. Lett., Volume 60, Number 1, Pages 189-194 (1997) on this subject.

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By way of background, a United States patent was recently issued covering the use of microwave irradiation in the Heck reaction. Based upon my research, the work of the alleged inventors appears to be at best concurrent with, and possibly later than, your work. We are investigating the validity of that patent, and any evidence of work done concurrently or earlier by others would be an important part of our evaluation.

Phil Summa

From: Phil Summa [phil@psumma.com]
Sent: Wednesday, April 04, 2001 2:53 PM
To: ibg@ipcl.sprintrpg.net.in
Subject: Facsimile Numbers

Dear Sirs:

I am trying to contact Drs. A. Wali, S. Muthukumar Pillai, and S. Satish of your company via facsimile. I have tried the following numbers (from your company website) without success:

91 265 375 165

91 265 370 040

Are these number correct or do I need to use a different number.

Thank you for your assistance.

Regards,
Phil Summa
13777 Ballantyne Corporate Place
Suite 315
Charlotte, NC 28277
USA
Telephone: 704-945-6701
Facsimile: Fax 704-945-6735
phil@psumma.com



*Innovators In
Microwave Technology*

International

3100 Smith Farm Road, P.O. Box 200, Matthews, NC 28106-0200 USA
P: 704-821-7015 * F: 704-821-7894 e: info@cem.com

April 9, 2001

Dr. Ravindranath
IPCL R&D Center
Vadodara, India

Dr. Ravindranath:

Your research center is using some CEM Corporation microwave equipment (in Dr. Kalpana's laboratory), and we are pleased that your work is going well. I have visited various IPCL facilities over the past several years during my travels to India. I recall doing a presentation to several persons at the R&D centre back in 1996, and discussing rapid ashing of polymers, digestion for elemental analysis, as well as fiber separation techniques. I do not recall discussing the Research areas, as we were mainly interested in process applications for microwave-based laboratory equipment.

We have reviewed with interest a scientific publication from your Research Centre at Vadodara, titled: "Heterogeneous Pd Catalysts and Microwave Irradiation in Heck Arylation", which is IPCL Communication No. 264. This was published by Elsevier Science B.V., Amsterdam, in React Kinet Catal Lett, Vol. 60, No. 1, 1997, I believe.

We are interested to know if there were earlier and similar IPCL Communications or publications related to your work with microwave irradiation.

Would it be possible to obtain a listing or bibliography of works that may have been published, or are available to the public sector? We try to keep abreast of the various areas where microwave equipment is used, so that we have a reference base for our scientists and engineers to be aware of in their continued development of our technology.



*Innovators In
Microwave Technology*

CEM Corporation developed the first commercially available microwave laboratory equipment over 25 years ago. We are the leaders in this technology, and are the largest manufacture of microwave laboratory equipment. We do our best to maintain leadership in the development of new products and accessories that can help our users in their work more effectively and efficiently.

Again, we appreciate your use of CEM Corporation equipment, and look forward to receiving any information you can provide. You may fax information to me at (1) 704-821-7894. Or, if you can attach to an e-mail: david.fish@cem.com

Sincerely,

A handwritten signature in cursive script, appearing to read 'David L. Fish'.

David L. Fish

Regional Manager, India

David Fish

05/03/01 08:10 AM

To: sm_pillai@yahoo.com @ Internet

cc:

Subject: Publication 294

Dr. Pillai:

Thank you for the e-mail message dated 4/19, regarding our discussions with Dr. Ravindranathan about IPCL publication 294, which is referenced in the published publication. I tried to call today, but the lines are constantly busy. As I indicated to Dr. Ravindranathan, we are trying to determine exactly when your work became available to others, either within the IPCL or to others outside IPCL.

There has been a 'claim' by others that they were the first to do the work that you have published as 'IPCL Communication 294'. We believe that the IPCL work was done several months prior to this claim, if not longer. Because of this, IPCL would then be considered as the organization that did it first.

In order for IPCL to be regarded as the first researchers in this area, it is important to have some type of time-dated document that would show that your work was, in fact, available to others before the actual 'publication date' in the Elsevier book, 1997. This publication shows 'Received October 17, 1995, as the first date. Obviously, the work was done earlier, before submittal.

We believe that "IPCL Communication 294" (the serial number that was assigned to this work and thus termed 'communication') was assigned to this material as a publication before the actual submittal date. This IPCL Communication 294 may have then been 'available' to others at IPCL, to read or what ever, in 1995, so that becomes the first date when this material was 'public disclosure'. Perhaps not to the 'general public', but at least to others at IPCL in the library, and that it was not 'confidential' material only to you and your co-authors.

If the material in IPCL Communication 294 was in fact available in your library for others to see, if they so chose, then the actual date that 294 was assigned becomes a critical issue.

Exactly when your studies became 'public disclosure', or available for others to read, becomes a very important issue, as we are trying to show that IPCL was, in fact, the first in the world to do this specific work in a microwave system. Yes - we have an interest in this, because a CEM system was used in this work. But, as the time requirements have expired, and a publication has been issued, there are no patentable rights obtainable by IPCL or others. Our intent it to dispute what others are claiming, and prove that IPCL was, in fact, the first to do this work.

If you can offer information that would show that IPCL Communication 294 was a document that was available in your library, before the actual publication date by Elsevier, this would be most helpful. Or - did you present this work to others at IPCL before the publication date as a seminar or document.

Thank you very much for your assistance in this matter. We look forward to your correspondence.

David L. Fish, Regional Manager, India
CEM Corporation
3100 Smith Farm Road
Matthews, NC USA 28106

FAX

Dr. S. Muthukumar Pillai
Research Centre, IPCL
Baroda-391346, INDIA
Email Id : <sm_pillai@yahoo.com>

24/05/2001

Ms. Cindy Moser
CEM Corporation
PO Box 200
3100, Smith Farm Road
Matthews
North Carolina 28106
USA

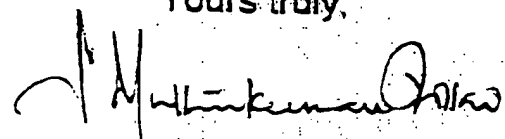
Fax No. 1 704 8217894

Sub: IPCL communication 294

Dear Ms. Cindy Moser,

Dr. David Fish was in contact with me about our work with above ref no. In my previous email to him I mentioned that we submitted our manuscript to Tetrahedron letters for publication. But the editor of the journal did not consider it suitable for the journal after consulting a referee. The point that emerges from this that we made the article for 'public disclosure' in the Feb - March 1995 itself. When I informed this to David Fish he asked me whether we can send the copy of correspondence. Herewith I am sending the papers for your reference. Hope they are useful to your lawyers for pleading your case. Kindly acknowledge the receipt of the papers and development in your effort in this case.


Yours truly,



Dr. S. Muthukumar Pillai

N.B: Total No. Pages 3




Summa & Allan, P.A.
11610 North Community House Road
Suite 200
Ballantyne Corporate Park
Charlotte, NC 28277
704-945-6700
Facsimile: 704-945-6735

CONFIRMATION

August 23, 2001

VIA FACSIMILE

Keith Warren, Esq.
Baron & Warren
18 South End
Kensington
London W8 5BU, ENGLAND

Re: Investigation of U.S. Patent No. 6,136,157; Our File 1700.104

Dear Keith:

This is a "toss up" request for yourself or any other appropriate member of your firm. As you are aware, we represent CEM Corporation in their intellectual property matters here in the United States, and you have assisted us with many of their European endeavors.

At the present time, CEM is faced with a competitor that holds a United States patent for a particular type of chemical reaction that uses microwave radiation as the initiating energy source. In investigating the validity of this patent, we have determined that earlier work was done in India on the exact same subject matter and even using a CEM microwave device. Thus, if this work can be established as meeting one or more of the criteria of § 102 of the U.S. patent statute, it will invalidate the competitor's patent.

To date, the best prior art that we have discovered is a journal article written by several scientists of the Indian Petrochemicals Corporation, Ltd. (IPCL), which apparently is one of the larger (or largest) chemical companies in India. This article was published at a date that is not sufficiently early to pre-date the relevant patent. On its face, however, the article states that it was also the subject of "IPCL Communication No. 294." According to informal information we have gathered, IPCL communication No. 294 predates the relevant patent filing date, and thus will anticipate and invalidate the relevant patent if it can be shown to qualify as a "printed publication" under § 102 of our patent law.

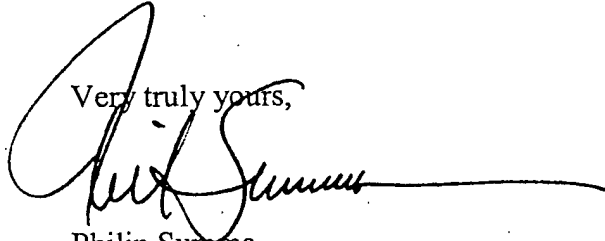
Although we are internally investigating the extent to which an IPCL communication can be considered a printed publication, we are also looking for independent confirmation of the same. Accordingly, I suggested to CEM that we enlist your help in identifying an appropriate attorney or firm in India who could independently confirm the public availability of this particular IPCL communication.

Keith Warren, Esq.
August 23, 2001
Page 2

Accordingly, do you have a suggestion for a lawyer or law firm for this task? We have assumed that because of the long-standing commercial relationships within the British Commonwealth, you would be able to give us the best recommendation.

Thank you for your help. If there is other information that we can or should provide to assist you in your recommendation, please let me know and we will make every attempt to forward it to you. If at all possible, we would like to confirm the public (or alternatively private) status of IPCL Communication No. 294 by mid-September.

Best regards.

Very truly yours,

Philip Summa

S:\FIRM DOCS\1700\104\Warren082201.doc



SUMMA & ALLAN P.A.
11610 NORTH COMMUNITY HOUSE ROAD
SUITE 200
BALLANTYNE CORPORATE PARK
CHARLOTTE NORTH CAROLINA 28277
PHONE 704-945-6700 FACSIMILE 704-945-6735
WWW.PSUMMA.COM

FACSIMILE TRANSMITTAL SHEET

TO:	FROM:
Keith Warren, Esq.	Philip Summa
COMPANY:	DATE:
Baron & Warren	August 23, 2001
FAX NUMBER:	TOTAL NO. OF PAGES INCLUDING COVER:
011-44-20-7937-4786	3
PHONE NUMBER:	SENDER'S REFERENCE NUMBER:
011-44-20-7937-0294	1700.104
RE:	YOUR REFERENCE NUMBER:
Investigation of U.S. Pat. No. 6,136,157	

☐ URGENT ☐ FOR REVIEW ☐ PLEASE COMMENT ☐ PLEASE REPLY ☐ PLEASE RECYCLE

NOTES/COMMENTS:

CONFIDENTIALITY NOTE

The information contained in this facsimile message is legally privileged and confidential. It is intended only for the use of the above-named individual or entity. If you are the reader of this message and are not the intended recipient, you are hereby notified that any dissemination, distribution, or copying of this facsimile is strictly prohibited. If you have received this facsimile in error, please immediately notify us by phone and return the original message to us at the address above via the mail. Thank you.

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Transmission

Transaction(s) completed

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SUMMA & ALLAN P.A.
11810 NORTH COMMUNITY HOUSE ROAD
SUITE 200

BALLANTYNE CORPORATE PARK
CHARLOTTE NORTH CAROLINA 28277
PHONE 704-945-6700 FACSIMILE 704-945-6735
WWW.PSUMMA.COM

FACSIMILE TRANSMITTAL SHEET

TO:

Keith Warren, Esq.

FROM:

Philip Summa

COMPANY:

Baron & Warren

DATE:

August 23, 2001

FAX NUMBER:

011-44-20-7937-4786

TOTAL NO. OF PAGES INCLUDING COVER:

3

PHONE NUMBER:

011-44-20-7937-0294

SENDER'S REFERENCE NUMBER:

1700.104

RE:

Investigation of U.S. Pat. No.
6,136,157

YOUR REFERENCE NUMBER:

☐ URGENT ☐ FOR REVIEW ☐ PLEASE COMMENT ☐ PLEASE REPLY ☐ PLEASE RECYCLE

NOTES/COMMENTS:

Baron & WarrenFax

To: Summa & Allan P.A.
 Attn: Philip Summa Esq
 Fax: 001 704 945 6735
 From: Keith Warren
 Date: 29 August, 2001
 Subject: Investigation of US Patent No. 6136157
 Your File: 1700.104

FILE

Page(s): 1 including this page

The contents of this transmission are privileged and confidential and intended solely for the use of the addressee. Any disclosure, distribution or copying of the contents, other than by the addressee, is strictly prohibited. If you receive this transmission in error, please notify us immediately and destroy the material received.

Dear Philip

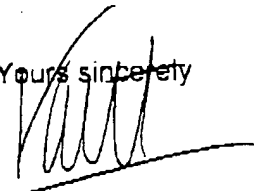
Replying to your letter of August 23 2001, I recommend that you enlist the assistance of the Indian firm of D.P. Ahuja & Co to help you with this investigation. A copy of their letter head giving details of the firm (including web site) is below.

We, ourselves, have primarily utilised the services of this firm for our Indian work since its formation some 25 years ago and have found them to be extremely competent and helpful.

If for some reason, they are unable to help you, please let me know and I will provide another recommendation.

Kindest regards

Yours sincerely



D. P. AHUJA (1924-1996)

S. D. AHUJA
 S. CHAKRABORTY
 S. H. GUPTA
 K. M. RAO
 J. VIJAY RAGHAVAN
 A. K. CHATTERJEE
 S. MUKHERJEE
 S. GHOSH
 I. S. BHATTACHARYA
 M. ISLAM
 A. DONG
 A. DHAR ROY
 P. K. MITRA
 S. ALANI
 K. GHOSH
 D. K. CHAUDHURY
 R. SIRCAR

D.P. AHUJA & Co.
 PATENT & TRADEMARK ATTORNEYS
 5J Syed Amir Ali Avenue Calcutta 700 019 INDIA

Telephone
 91 (33) 2401511
 91 (33) 2473158
 Telefax
 91 (33) 4757524
 91 (33) 2478982

BANGALORE MADRAS NEW DELHI

Email
 dpahuja@vsnl.com
 Website
 www.dpahuja.com

INDIA
 AFGHANISTAN
 BANGLADESH
 BHUTAN
 INDONESIA
 NEPAL
 PAKISTAN
 SRILANKA

Patent & Trade Mark Attorneys
 18 South End, Kensington, London, W8 5BU
 Tel: +44 (0)20 7937 0294 Fax: +44 (0)20 7937 4786
 E-Mail: patags@baron-warren.co.uk

D. P. Ahuja & Co.

HEAD OFFICE

53 Syed Amir Ali Avenue
Fourth Floor
Calcutta 700019
INDIA

PH.: 011-91-33-401511
2473158

FAX: 011-91-33-4757524
2485229

Alternate Fax: 011-91-33-2478982

BRANCH OFFICE

D-4, Casa Lavelle No. 1
84/85 Lavelle Road
Bangalore 560001
INDIA

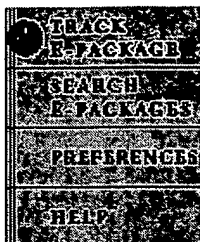
PH.: 011-91-33-401511
FAX: 011-91-33-757524



UPS ONLINE COURIER

PREPARE E-PACKAGE	TRACK E-PACKAGE	ACCOUNT	BILLING	ADDRESS BOOK	LOG OFF
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Delivery Detail


**Recipient(s)**

The recipient has not yet been notified.

dpahuja@vsnl.com

Delivery notification pending.

OnLine Courier® e-Package [Cancel this delivery](#)**Subject:** Request for Services**Package:** 1 file(s)**Files:** [Ahuja083001.DOC](#) 27 KB Microsoft Word Document**Sent:** 08/30/2001 at 15:01:24**Destination:** Far East (Asia)**Scheduled:** 08/30/2001 at 15:01:23**Expires:** 09/06/2001 at 15:01:24**Delivery:** yes**Billing:** 1700.104**Confirmation:****Code:****Security:** Secure connection. Server encryption. No password.*Powered By Tumbleweed*



Summa & Allan, P.A.
11610 North Community House Road
Suite 200
Ballantyne Corporate Park
Charlotte, NC 28277
704-945-6700
Facsimile: 704-945-6735

VIA ELECTRONIC COURIER

August 30, 2001

D. P. Ahuja & co.
P.O. Box 16033
Calcutta 7000 019
INDIA

Re: Potential Representation of CEM Corporation; Our File 1700.104

Dear Sirs:

Upon recommendation of Keith Warren, we are writing to request your potential assistance in India with certain matters related to the validity of a U.S. Patent. By way of introduction, we are also a specialized patent firm and we invite you to peruse our web site at www.psumma.com or our profile in Martindale-Hubbell ([Martindale-Hubbell@Lawyer Locator](http://Martindale-Hubbell.com)). Before turning to the merits of our request, we need to make sure that our client and its work present you with no conflict. Thus, our client is CEM Corporation of Matthews, North Carolina, USA. CEM is the world's leading designer and manufacturer of laboratory equipment that incorporates the use of microwave energy to heat samples. Further information about CEM and their products can be obtained from their website at <http://www.cem.com/>.

Would you please let us know immediately whether carrying out work on behalf of CEM would present you with any conflict. Once we can confirm that conflicts can be avoided, we will forward our particular requests for services.

Thank you for your assistance.

Very truly yours,

Philip Summa

S:\FIRM DOCS\1700\104\Ahuja083001.DOC



UPS ONLINE COURIER

PREPARE
E-PACKAGETRACK
E-PACKAGE

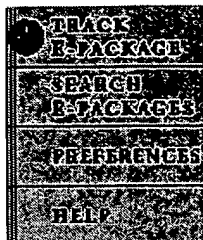
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VIA ELECTRONIC COURIER

September 6, 2001

D. P. Ahuja & Co.
P.O. Box 16033
Calcutta 7000 019
INDIA

Re: Investigation of U.S. Patent No. 6,136,157; Our File 1700.104

Dear Sirs:

Thank you for your prompt response to our conflict inquiry. Our specific request is as follows.

Background

Our client CEM is faced with a competitor that holds the above-referenced United States patent for a particular type of chemical reaction that uses microwave radiation as the initiating energy source. A copy of the '157 patent in .pdf format is forwarded herewith. In investigating the validity of this patent, we have determined that earlier work was done in India on the exact same subject matter using a CEM microwave device. Thus, if this work can be established as meeting one or more of the criteria of § 102 of the U.S. patent statute, it will invalidate the competitor's patent.

To date, the best prior art that we have discovered is a journal article written by several scientists of the Indian Petrochemicals Corporation, Ltd. (IPCL): Wali, *Heterogeneous Pd Catalysts and Microwave Irradiation in Heck Arylation*, React. Kinet. Catal. Lett. Vol. 60, No. 1, 189-194 (1997). This article does not pre-date the '157 patent. On its face, however, the article states that it was also the subject of "IPCL Communication No. 294." According to informal information we have gathered, IPCL Communication No. 294 predates the patent filing date, and thus will anticipate and invalidate the patent if it can be shown to qualify as a "printed publication" under § 102 of U.S. patent law.

Although we are internally investigating the extent to which an IPCL Communication can be considered a printed publication, we are also looking for independent confirmation of

D. P. Ahuja & Co.
September 6, 2001
Page 2

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We need to know three things about IPCL Communication No. 294:

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- (2) Its date; and
- (3) Whether it was available to the public (even if the public never bothered to seek it out.)

Thank you for your help. If there is other information that we can or should provide to assist you, please let me know and we will make every attempt to forward it to you. If at all possible, we would like to confirm the public (or alternatively private) status of IPCL Communication No. 294 by mid-September.

Best regards.

Very truly yours,

Philip Summa


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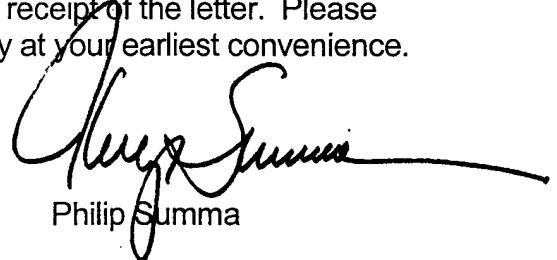
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D. P. Ahuja & Co.
September 6, 2001
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Best regards.

Very truly yours,

Philip Summa

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D. P. AHUJA † (1924-1996)

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D.P. AHUJA & Co.
PATENT & TRADEMARK ATTORNEYS
53 Syed Amir Ali Avenue Calcutta 700 019 INDIA

Telephone

91 (33) 2401511

91 (33) 2473158

Telefax

91 (33) 4757524

91 (33) 2478982

BANGALORE MADRAS NEWDELHI

Email

dpahuja@vsnl.com

Website

www.dpahuja.com

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Summa & Allen, P. A.
11610 North Community House Road
Suite 200
Ballantyne Corporate Park
Charlotte, NC 28277
USA

2001 September 12

Our Ref.: PGF1786.1716.SDA

Your Ref.: 1700.104

Attn.: Philip Summa

Fax: 1 704 945 6735

Dear Sirs,

Re.: Invalidation of US Patent

Thank you for your letter of 6 September, 2001 concerning the above matter.

Our charges for attempting to search and locate the literature (IPCL communication no. 294), or any other material that will assist invalidation of US Patent No. 6,136, 157, would be USD750. Should we succeed in locating the article or literature and provide you all the details you've asked for, our charges, depending on the extent of advanced searches we would need to conduct, can go upto USD1000.

In other words, cost of search would be USD750, and that of a successful search would be USD1000 approximately. Please confirm if we should go ahead.

We shall need a copy of the article by Wali {*Heterogeneous Pd Catalysts and Microwave Irradiation in Heck Arylation*, React. Kinet. Catal. Lett. Vol. 60, No. 1, 189-194 (1997)}, and a copy of the front page of the journal.

Very truly yours,


D. P. AHUJA & CO.

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VIA FACSIMILE

September 12, 2001

D. P. Ahuja & Co.
P.O. Box 16033
Calcutta 7000 019
INDIA

Re: Investigation of U.S. Patent No. 6,136,157; Our File 1700.104

Dear Sirs:

Thank you for your letter of September 12. The fees and associated costs that you describe are acceptable, and you are hereby authorized to proceed with your search.

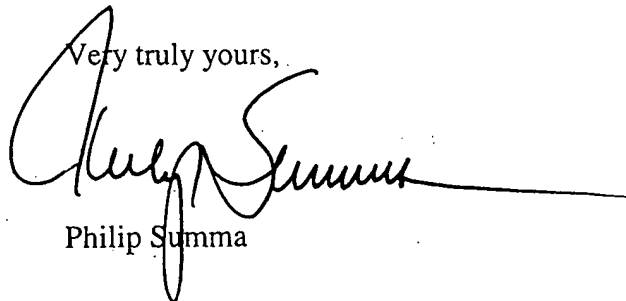
A copy of the Wali reference is transmitted herewith. We do not have a copy of the front page of the journal, but you will note that some of the publication information is listed on the top of the first page of the article.

To repeat, our goal is to establish that Wali's earlier submission of this work, in the form of IPCL Communication No. 294, anticipates the claims of the '157 patent. To do so we need to learn the contents of IPCL Communication No. 294 (preferably by seeing an exact copy), whether it was available to the public, and the date on which it became available.

Thank you for your assistance.

Best regards.

Very truly yours,


Philip Summa

Enclosures

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RKCL2873

HETEROGENEOUS Pd CATALYSTS AND MICROWAVE IRRADIATION IN HECK ARYLATION*

A. Wali, S. Muthukumar Pillai and S. Satish
Research Centre, Indian Petrochemicals Corporation Ltd.,
Vadodara-391 346, India

Received October 17, 1995

Accepted March 19, 1996

Abstract

Pd metal dispersed on supports like γ - Al_2O_3 , C, MgO and CaCO_3 is an efficient and recyclable catalyst in Heck arylation of several olefins. This reaction is also effective in microwave environment.

Keywords: Heck arylation, supported palladium, microwave irradiation

INTRODUCTION

The Pd(0)-catalyzed reaction of an aryl or vinyl halide with an alkene known as the Heck reaction is an important method of C-C bond formation [1]. Pd(OAc)₂ is generally employed as a soluble catalyst in this organometallic reaction [2]. However, a variety of such reactions are favorably amenable to catalysis by dispersed metals. The heterogeneous catalysts in synthetically useful reactions offer significant economic, practical and environmental advantages [3]. We present here a few Heck arylations using various dispersed Pd systems including the novel Pd/MgO and Pd/SiO₂-Al₂O₃ catalyst. Besides, the use of microwave irradiation in such a reaction is being reported, to our knowledge, for the first time.

* IPCL Communication No. 294

EXPERIMENTAL

The various supported catalysts were prepared by standard impregnation techniques using $\text{Pd}(\text{acac})_2$ as precursor. These catalysts were then reduced with H_2 at 100°C . In general, the products were characterized by the usual spectral methods and found in agreement with the reported values.

A typical procedure is described for the preparation of cinnamionitrile (1): A mixture of iodobenzene (2.04 g, 0.01 mole), acrylonitrile (0.53 g, 0.01 mol), 5% Pd/C (0.212 g), PPh_3 (0.078 g), Et_3N (1 g) and CH_3CN (10 mL) were taken in a 160 mL Parr autoclave under N_2 (25 psig). The mixture was heated at 140°C for 14 h. After cooling, the reaction mixture was filtered, concentrated and column chromatographed over silica gel using petroleum ether ($40-60^\circ\text{C}$) as eluent to give cinnamionitrile as a mixture of E and Z isomers (0.80 g). The spectral data of the product matched with the reported values [4]. The yield and selectivity values given in Table 1 are derived from GC analysis (1.5% OV-17) using the internal standard method.

Table 1
Arylation of acrylonitrile over various Pd catalysts*

Catalyst	Yield (%) ^b	E : Z isomers of	1	2	Selectivity
$\text{Pd}(\text{acac})_2$	70.4	82:18			100
5 % Pd/C	62	77:23			100
5% $\text{Pd}/\gamma\text{-Al}_2\text{O}_3$	72.4	77:23			95.4
5% Pd/MgO	77.8	78:22			91.3
5% Pd/CaCO_3	59.3	78:22			96.5
0.6% $\text{Pd}/\gamma\text{-Al}_2\text{O}_3$		No reaction			
0.3 % $\text{Pd}/\text{SiO}_2\text{-Al}_2\text{O}_3$	55.2	78:22			100
0.3 % $\text{Pd}/\gamma\text{-Al}_2\text{O}_3$	86.0	74:26			96.8

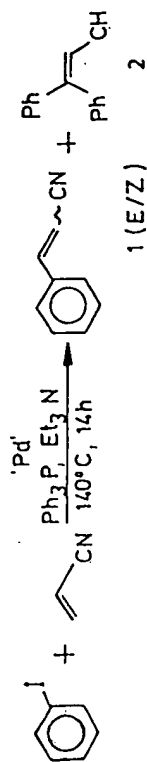
* $\text{Pd}:\text{ACN} = 1:100$ (mol/mol); $\text{CH}_3\text{CN} = \text{solvent}$; 140°C ; 14h;

$\text{PhI}:\text{ACN} = 1:1$; $\text{Et}_3\text{N}:\text{ACN} = 1:1$; $\text{PPh}_3/\text{Pd} = 3$. All the supported catalysts are reduced before use.

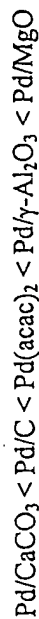
^bBased on ACN

Microwave experiments were conducted in CEM MDS 2000 instrument at 80% power. The machine had a magnetron frequency of 455 MHz and a maximum output of 650 W.

RESULTS AND DISCUSSION



Several Pd catalysts have been examined in the reaction of acrylonitrile (ACN) with iodobenzene (Table 1). E and Z isomers of cinnamionitrile (1) are smoothly obtained in a single step. 1-Cyano-2,2-diphenylethene (2) is also formed in varying proportion depending upon reaction conditions. The activity of the catalysts follows the series:



Among the 5% loaded catalysts the yield of product is maximum on Pd/MgO . Dossi *et al.* have reported that in chloride-free Pd/MgO catalysts electron transfer from the strongly basic O^{2-} surface ion to metal surface leads to 'anchoring' of the metal to support [5]. This prompts higher activity of Pd/MgO catalyst.

However, lower loading of Pd results in higher activity as seen in case of 0.3% $\text{Pd}/\gamma\text{-Al}_2\text{O}_3$. Interestingly, the ratio of E and Z isomers of 1 formed over supported Pd catalysts is distinct from the homogeneous counterpart. Further, 2 is formed as byproduct over Pd dispersed on amphoteric Al_2O_3 and basic MgO and CaCO_3 supports.

To establish some generality of dispersed Pd catalysis in Heck arylation, iodobenzene, *o*-iodoanisole and benzoyl chloride were used as arylating agents for several olefins using 5% Pd/C . As expected, the arylation takes place smoothly with these substrates (Table 2). However, *o*-iodonitrobenzene was inactive under these conditions.

The reduced Pd/MgO was recycled in several runs for arylation of acrylonitrile to examine the stability and life of the catalyst (Table 3). ESCA investigations of fresh and recycled Pd/MgO catalysts show that Pd is anchored on MgO . The atomic absorption spectral analysis of the liquid product after a run shows that only a small amount (0.13%) of loaded Pd is leached out, thereby ruling out the involvement of Pd species in the liquid phase as catalyst. The formation of products in the recycle runs by the same Pd/MgO suggests that the reaction is indeed promoted by Pd metal dispersed on the support.

activity of the catalyst in the second recycle. This is due to gradual masking of Pd sites with hydrocarbons and generation of non-reducible Pd²⁺ sites.

Table 4

Microwave induced Heck reaction of iodobenzene and 1-decene

Catalyst	Time (min) ^a	Conversion to phenyldecene ^b
5 % Pd/C	10.5	53.7
5 % Pd/MgO	10.0	39.2
5 % Pd/ γ -Al ₂ O ₃	10.0	58.6
0.3 % Pd/SiO ₂ -Al ₂ O ₃	6.0	53.5

^aCaution : explosive pressure can build up

^banalyzed by GC

In the present study the influence of microwave irradiation was examined to improve the yield and decrease the reaction time. Accordingly, the Heck reaction was designed for high boiling substrates and experiments were conducted in a microwave digester using supported catalysts (Table 4). The comparable activity of the catalysts in nearly the same conditions is discernible besides attesting to the applicability of microwave in Heck arylation.

Acknowledgement. We thank IPCL management for permission to publish this work, Dr. V.K. Kaushik for ESCA studies, and P.J. Patel and A.B. Parikh for technical assistance.

REFERENCES

1. a) R.F. Heck: *Acc. Chem. Res.*, 12, 146 (1979);
b) C.M. Andersen, A. Hallberg: *J. Org. Chem.*, 53, 235 (1988);
c) G.D. Davis, Jr.: *Chem. Rev.*, 89, 1433 (1989);
d) A. de Meijere, F.E. Meyer: *Angew. Chem. Int. Ed. Engl.*, 33, 2379 (1994).
2. a) A. Hallberg, L. Westfelt, B. Holm: *J. Org. Chem.*, 46, 5414 (1981);
b) R.C. Larock, W.H. Gong, B.E. Baker: *Tetrahedron Lett.*, 2603 (1989);
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Table 2

Heck arylations using 5 % Pd/C^a

Iodoarene	Olefin	Products	Yield (%)
<i>o</i> -Iodoanisole	Styrene	2-Methoxy stilbene	71
Iodobenzene	Styrene	Stilbene (E)	55
<i>o</i> -Iodoanisole ^b	Ethylene	2-Methoxystyrene	20
		2,2'-Dimethoxy stilbene	7
Iodobenzene	Ethylene	Styrene	30
		Stilbene	17
Iodobenzene	α -Methyl styrene	α -Methyl stilbene	55
<i>o</i> -Iodoanisole	α -Methyl styrene	α -Methyl-2-methoxy stilbene	45
Iodobenzene	1-Decene	Phenyldecene ^c	71
Iodobenzene	Acrylonitrile	Cinnomonitrile (Z & E)	62
Benzoyl chloride	Acrylonitrile	Cinnomonitrile (Z & E) ^d	44
Iodobenzene	α -Methylacrylonitrile	α -Methylcinnamonitrile (Z & E)	31

^aCH₃CN = solvent; 140°C; 14 h, isolated yield.

^b140°C; 8 h

^cIsomeric mixture

^dBenzene solvent, 100°C

Table 3

Activity of Pd/MgO and the effect of recycles on the arylation of acrylonitrile with iodobenzene^a

Run	Time (h)	Pd:ACN (mol/mol)	Efficiency ^b	E : Z	Selectivity
				1	1 2
Fresh catalyst	10	1:100	71.2	78:22	97.7 2.3
1st recycle	10	1:200	133.6	77:23	97.5 2.5
2nd recycle	10	1:200	27.0	78:22	100 -

^aconditions as in Table 1

^befficiency = mole of (1+2) / g atom of Pd

Apparently, there is an increase in activity of the catalyst during the first recycle run. An increase in Pd(3d_{5/2})/Mg(2s) intensity ratio of photoelectron lines is observed with the first recycle catalyst compared to reduced catalyst. Further HI formed in the reaction is neutralized by MgO. This additional role of MgO is a key factor in higher activity of first recycle catalyst. Then there is a decrease in

4. A. Spencer: *J. Organomet. Chem.*, **240**, 209 (1982), and references therein.
5. C. Dossi, A. Fusi, S. Recchia, M. Anghileri, R. Psaro: *J. Chem. Soc., Chem. Commun.*, **1245** (1994).

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React. Kinet. Catal. Lett.
Vol. 60, No. 1, 195-203
(1997)

RKCL3088

STUDY ON THE REACTIVITY OF In_2O_3 IN MIXTURES WITH AMMONIUM-ZEOLITES

R.M. Mihályi^a, G. Pál-Borbély^a, H.K. Beyer^a, Ch. Minchev^b, Y. Neinska^b
and H.G. Karge^c

^aCentral Research Institute of Chemistry, Hungarian Academy of Sciences,
Pusztaszeri út 59-67, 1025 Budapest, Hungary.

^bInstitute of Organic Chemistry, Bulgarian Academy of Sciences, Sofia, Bulgaria,

^cFritz Haber Institute of the Max Planck Society, Faradayweg 4-6, 14195 Berlin,
Germany

Received January 8, 1997

Accepted January 16, 1997

Abstract

In ground mixtures of In_2O_3 and NH_4Y , incorporation of In^+ cations into the zeolitic phase occurs upon thermal treatment by partial reductive solid-state ion exchange associated with oxidation of ammonium ions or released ammonia to N_2 and NH_2OH . Cationic InO^+ species, created in zeolites by reductive solid-state ion exchange of $\text{In}_2\text{O}_3/\text{NH}_4$ -zeolite mixtures in hydrogen atmosphere and subsequent oxidation of the In^+ lattice cations by oxygen, do not undergo autoreduction up to 970 K. Reductive solid-state ion exchange easily proceeds in carbon monoxide atmosphere at temperatures between 620 and 770 K. The significance of these observations for the use of indium-containing zeolites as catalysts is discussed.

Keywords: $\text{In}(\text{I})$ -Y zeolite, reductive solid-state ion exchange, extra-framework oxygen, redox behavior of In -Y zeolites

INTRODUCTION

There has been considerable incentive for several decades to find a catalyst for the decomposition of NO in order to remove harmful NO from the exhaust of various combustion sources. Since copper-exchanged zeolites, especially those with MFI structure, proved to be active catalysts for the direct decomposition of

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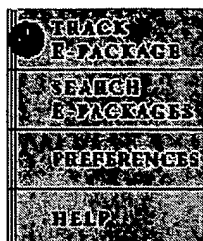
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Summa & Allan, P.A.
11610 North Community House Road
Suite 200
Ballantyne Corporate Park
Charlotte, NC 28277
704-945-6700
Facsimile: 704-945-6735

VIA ELECTRONIC COURIER

December 10, 2001

D. P. Ahuja & Co.
P.O. Box 16033
Calcutta 7000 019
INDIA

Re: Investigation of U.S. Patent No. 6,136,157; Our File 1700.104

Dear Sirs:

By our letters of September 6, 2001 and September 12, 2001, we requested your assistance in investigating the following article:

Wali, *Heterogeneous Pd Catalysts and Microwave Irradiation in Heck Arylation*, React. Kinet. Catal. Lett. Vol. 60, No. 1, 189-194 (1997).

We asked for your response by mid-September if at all possible.

To date, we have received no response whatsoever. Can you complete this request before December 31? If you cannot commit to doing so, we will need to seek other assistance.

Very truly yours,


Philip Summa

**SUMMA &
ALLAN, P.A.**
PATENT ATTORNEYS

11610 NORTH COMMUNITY HOUSE RD.
SUITE 200
BALLANTYNE CORPORATE PARK
CHARLOTTE, NC 28277
T: 704.945.6700
F: 704.945.6735
W: WWW.PSUMMA.COM

VIA DHL

January 11, 2002

 D. P. Ahuja & Co.
P.O. Box 16033
Calcutta 7000 019
INDIA

Re: Investigation of U.S. Patent No. 6,136,157; Our File 1700.104

Dear Sirs:

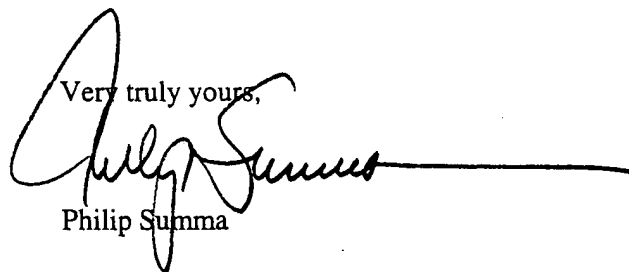
By our letters of September 6, 2001 and September 12, 2001, we requested your assistance in investigating the following article:

Wali, *Heterogeneous Pd Catalysts and Microwave Irradiation in Heck Arylation*, React. Kinet. Catal. Lett. Vol. 60, No. 1, 189-194 (1997).

We asked for your response by mid-September if at all possible.

To date, we have received no response whatsoever. Facsimile messages have gone unanswered and you have not replied to letters sent via electronic courier to your listed e-mail address. Can you complete our original request? Is there something lacking that you need before you can complete the requested task? We and our client are most disturbed by this unreasonably long delay and lack of communication. If you cannot commit to finishing this project by January 31, we will be forced to seek other assistance.

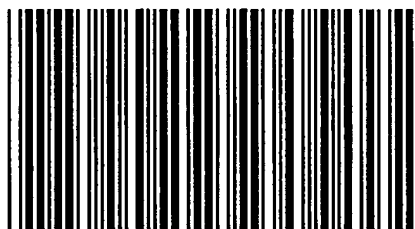
Very truly yours,


Philip Summa



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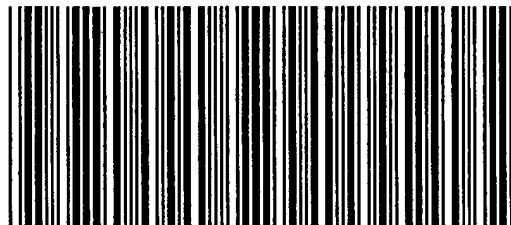
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700 019 India
Ph: 91-33-2401511 Fax: 91-33-4757524

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D.P. AHUJA & Co.
PATENT & TRADEMARK ATTORNEYS
53 Syed Amir Ali Avenue Calcutta 700 019 INDIA
Telephone
91 (33) 2401511
91 (33) 2473158
Telefax
91 (33) 4757524
91 (33) 2478982
BANGALORE MADRAS NEWDELHI
Email
dpahuja@vsnl.com
Website
www.dpahuja.com

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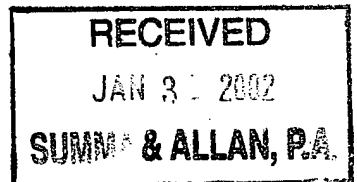
Summa & Allan, P. A.
11610 North Community House Road
Suite 200
Ballantyne Corporate Park
Charlotte, NC 28277
USA

2002.January 16

Our Ref.: PGF1786.1828.SDA
Your Ref.: 1700.104
Attn.: Philip Summa
Fax: 1 704 945 6735

Dear Sirs,

Re.: Investigation of US Patent No. 6,136,157



Thank you for your letter of 11 January, 2002 concerning the above.

We have not been able to report to you earlier because the investigation took more time than we had anticipated. However, we have now nearly completed the investigation and hope to sent you our report within this month.

Please note we have not received any communication from you after 13 September, 2001 when we received your faxed letter of 12 September. For your ready reference our contact address is: -

D. P. Ahuja & Co.
53 Syed Amir Ali Avenue
Fourth Floor
Calcutta - 700 019
Fax # +91 33 4757524/2478982

E-mail: dpahuja@vsnl.com

Very truly yours,

D. P. Ahuja & Co.
D. P. AHUJA & CO.

D. P. AHUJA ‡ (1924-1996)

S. D. AHUJA
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S. R. GUPTA
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D.P. AHUJA & Co.
PATENT & TRADEMARK ATTORNEYS
53 Syed Amir Ali Avenue Calcutta 700 019 INDIA
Telephone
91 (33) 2401511
91 (33) 2473158
Telefax
91 (33) 4757514
91 (33) 2478982
BANGALORE MADRAS NEWDELHI
Email
dpahuja@vsnl.com
Website
www.dpahuja.com

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Summa & Allen, P. A.
11610 North Community House Road
Suite 200
Ballantyne Corporate Park
Charlotte, NC 28277
USA

2002 February 01

Our Ref.: PSG1122.001.SSM
Your Ref.: 1700.104
Attn.: Philip Summa
Fax: 1 704 945 6735

Dear Sirs,

Re.: Investigation of US Patent No. 6,136,157. Investigation

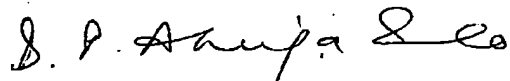
This is further to our letter of 16 January, 2002 on the above.

We have established contact with the Research and Development division of Indian Petrochemicals Corporation Ltd. in Vadodara in Gujarat. Of the three co-writers of the paper, "Heterogeneous Pd Catalysts and Microwave Irradiation in Heck Arylation", A. Wali and S. Satish have left the company. Mr. S. Muthukumar Pillai is still with IPCL, now as Manager - R&D. We have arranged to meet S. M. Pillai on Monday, 4 February, and hope to be able to obtain the required information.

Nobody at the offices of IPCL in New Delhi and Ahmedabad seems to be aware of the existence of any in-house journal, research papers, etc. (IPCL Communications No. 294). This is because IPCL Communications has very restrictive circulation, and meant for the R&D Division of IPCL only.

As indicated earlier, this assignment is taking much longer time than we had envisaged because of the strong reluctance of IPCL and related persons to discuss any matter pertaining to its research and research scientists. However, we should succeed in our endeavour to engage S. M. Pillai and/or his present associates working in the R&D Division of IPCL in Vadodara and revert with more information early next week. Trust this is in order.

Very truly yours,



D. P. AHUJA & CO.

Phil Summa

Fr m: cindy.moser@cem.com
Sent: Tuesday, April 09, 2002 9:43 AM
To: phil@psumma.com
Subject: Re: Former IPCL work



FILE0001.HTM

Cindy Moser

----- Forwarded by Cindy Moser/CEM on 04/09/02 09:34 AM -----

David Fish
02/07/02 02:39 PM

To: Cindy Moser/CEM@CEM
cc:
Subject: Re: Former IPCL work

In this 'file' is Satish's address. I don't know what the "OPCW" is. He may be in some type of UN agency.

I am in receipt of your e mail and my contace address is as follows:
Dr S. Satish, OPCW, Johan De Wittlaan 32, 2517 JR, Den Haag, The Netherlands.
Sincerely,
Satish

----- Forwarded by David Fish/CEM on 02/07/02 02:38 PM -----

sheosatish@hotmail.com
02/06/02 11:37 AM

To: David Fish@CEM
cc:
Subject: Re: Former IPCL work

Phil Summa

From: cindy.moser@cem.com
Sent: Tuesday, April 09, 2002 9:43 AM
To: phil@psumma.com
Subject: Re: addresses

Cindy Moser
----- Forwarded by Cindy Moser/CEM on 04/09/02 09:33 AM -----

David Fish
02/05/02 09:10 AM

To: Cindy Moser/CEM@CEM
cc:
Subject: Re: addresses

These people also live in housing apartments in the "IPCL Housing Compound", adjacent to the petrochemicals complex, but they were 'reluctant' to provide their residence addresses. They said that communication regarding IPCL should be directed to them at their office at IPCL. This P/C complex has 21 production operating units, each with different end products, and covers several square miles of area.

Dr. S. Muthukumar Pillai
Manager (R&D)
Indian Petrochemicals Corporation Limited
PO: Petrochemicals - 391 346
Dist: Vadodara
India

Residence phone: 011-91-265-266-5480

Office: 011-91-265-272-011/031/711 Extension 3066
Fax: 011-91-265-272-098
E: sm_pillai@yahoo.com

Dr. A. Wali
Scientist
Indian Petrochemicals Corporation Limited
same.....

Office: same as above

Sheo Satish, Netherlands someplace

sheosatish@hotmail.com

Cindy Moser
02/04/02 02:28 PM

To: David Fish/CEM@CEM
cc:
Subject: addresses

David,
Did you get the residence addresses for Wali & Pillai while you were in India?
Thanks,
Cindy Moser



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5/8/02 Checked status – same as above
5/9/02 Checked status – same as above
5/10/02 Checked status – same as above
5/13/02 Checked status – same as above (see attached)